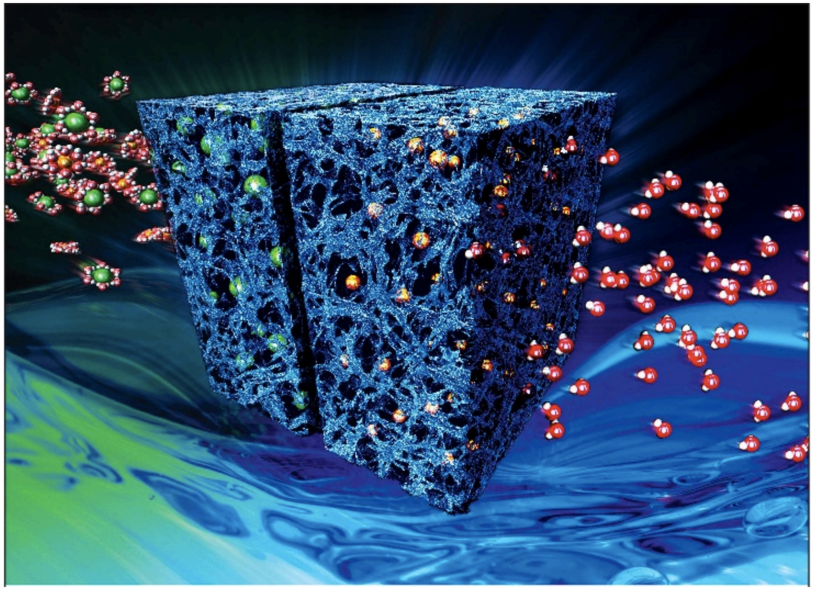
STANFORD MICROFLUIDICS LABORATORY

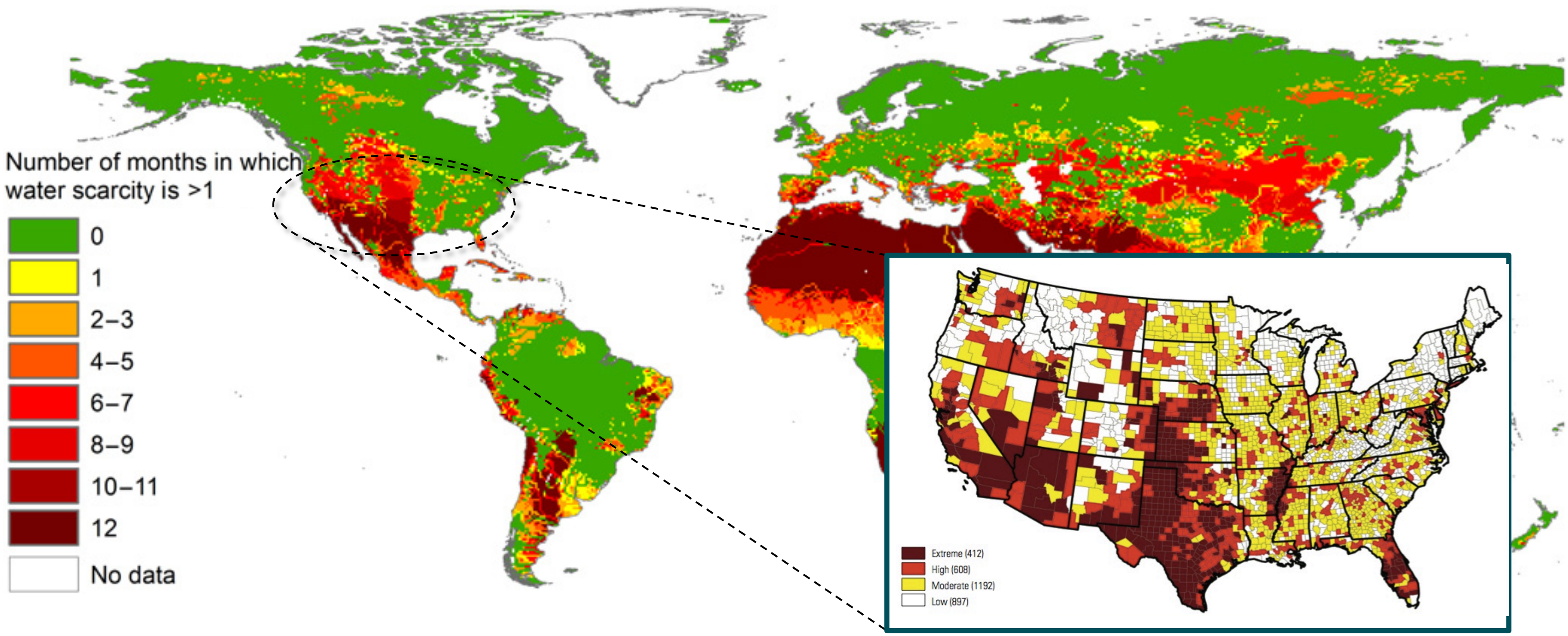
Capacitive Deionization (CDI) Cell Fabrication with *Voltera V-One* Paste Dispenser



DIEGO A. HUYKE AND DIEGO I. OYARZUN

Swaroop Kommera (Staff) and Mark Zdeblick (External)

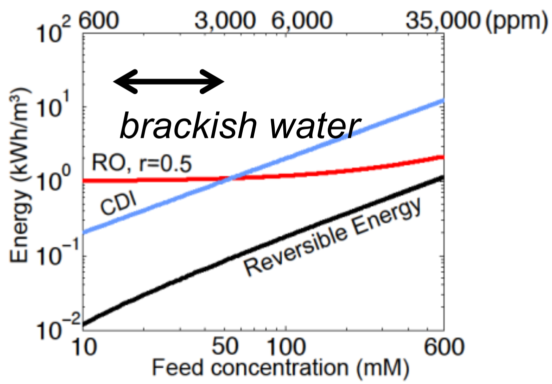
Fresh Water Is Becoming Scarce



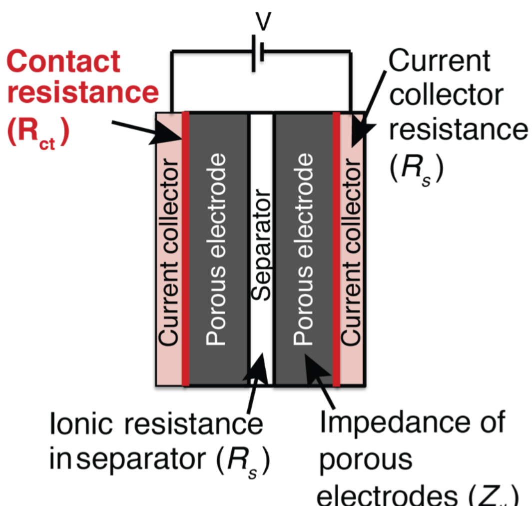
- Four billion people experience *severe* water scarcity > 1 month/year (Mekonnen et al., Sci. Adv., 2, 2016)
- *Brackish* water accounts for 80% of US desalination market

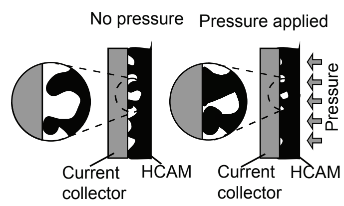
What is Capacitive Deionization (CDI)?

Energy Efficiency of CDI (and Improvements)

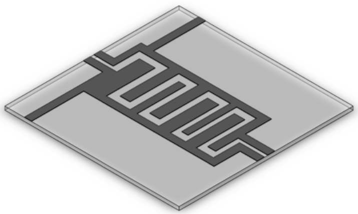


Projected: ~0.2 kWh/m^3
(~5x better than RO)

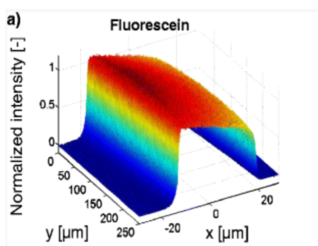




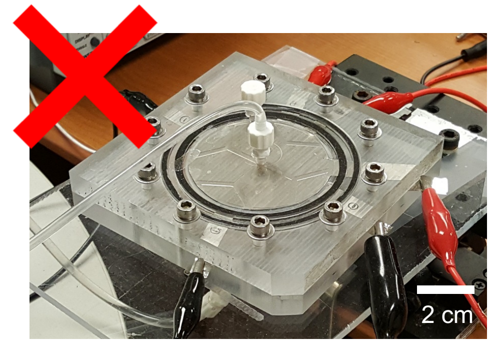
Contact resistance



Ion diffusion

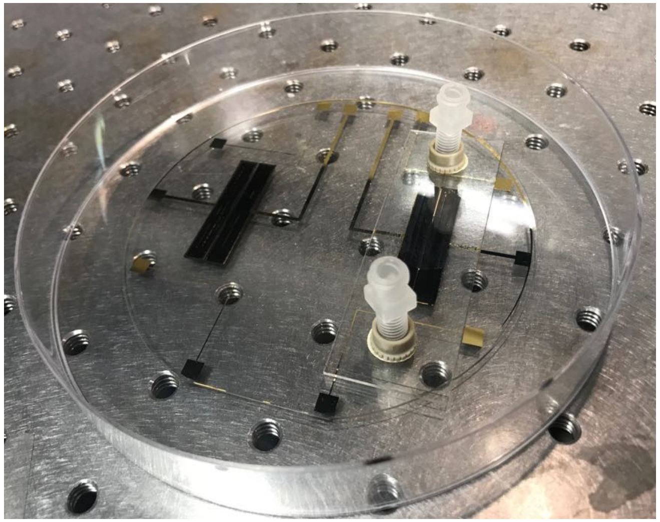


Understanding

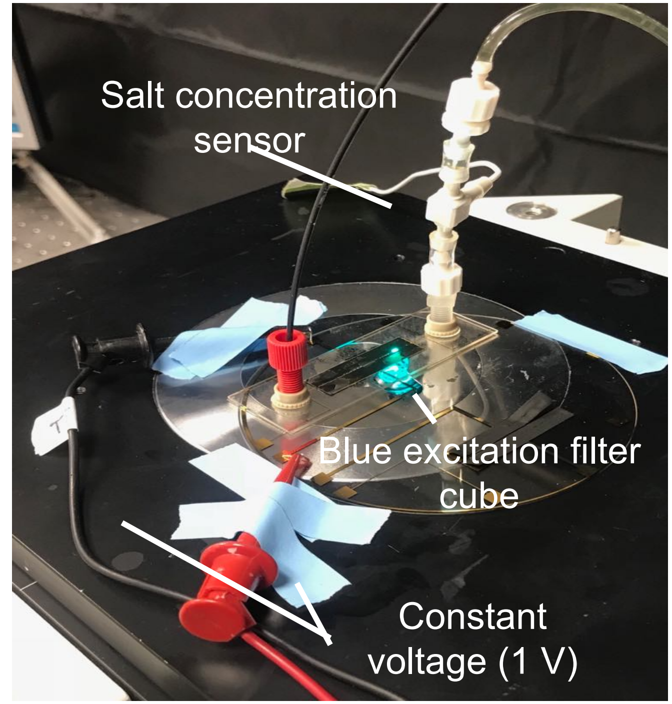


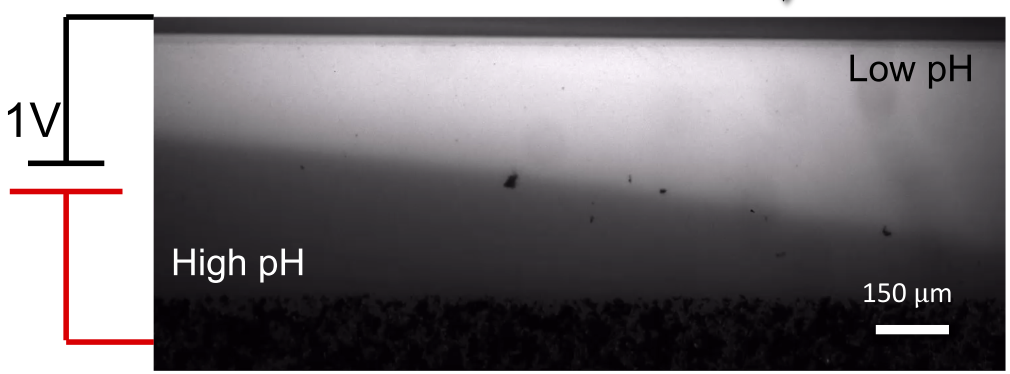
Activated carbon (AC) slurry

Microfabricated CDI Cell



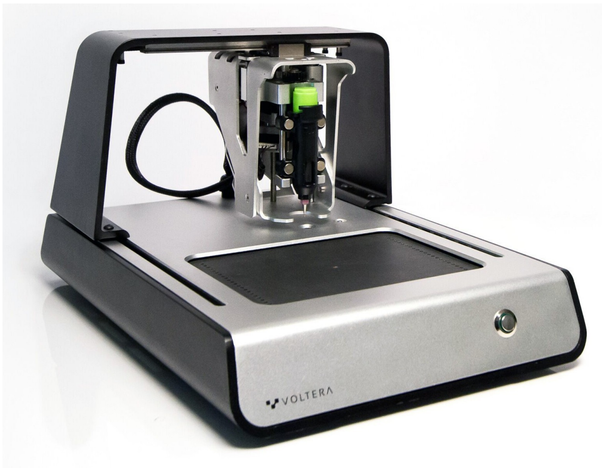
Epifluorescence microscopy

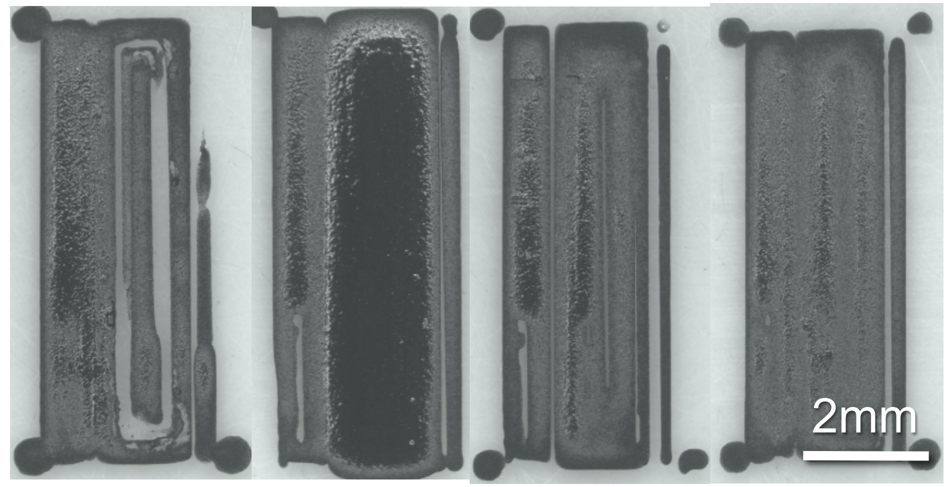


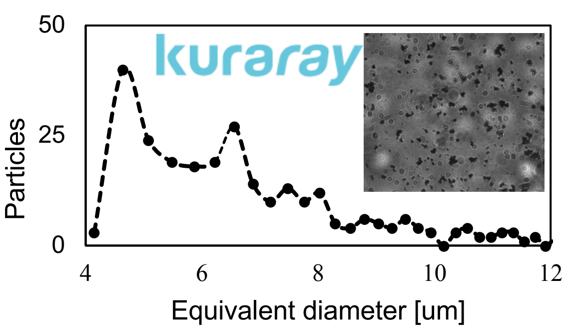


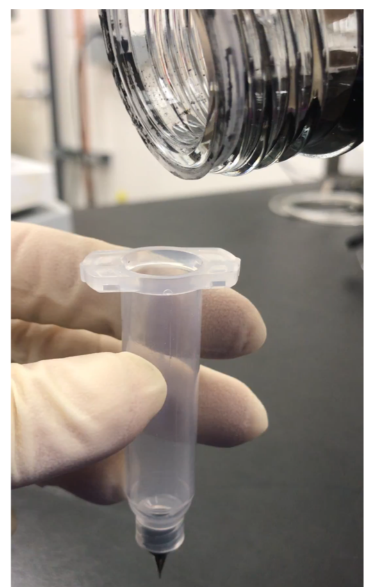
- Feed: KCl 20mM + Fluorescein 40µM
- pH effects on AC surface
- Min gap between electrodes

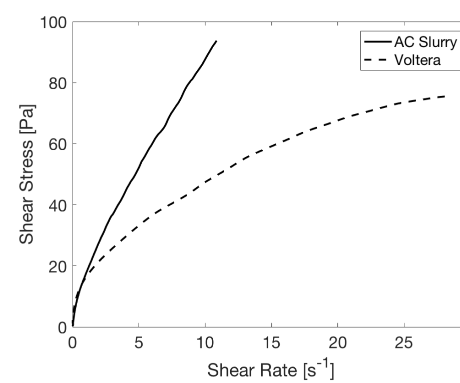
Voltera V-One





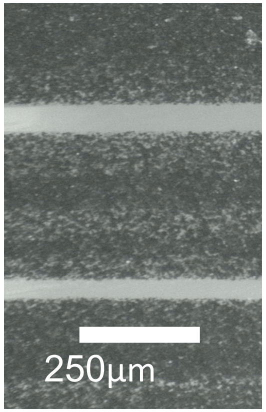


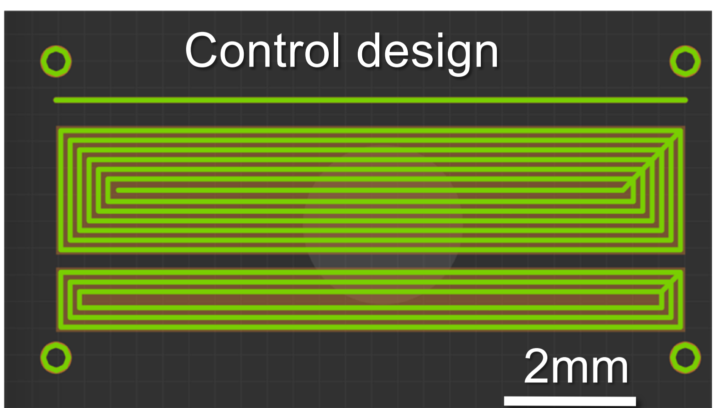


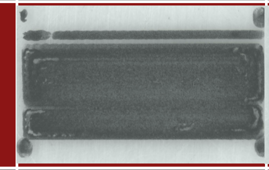
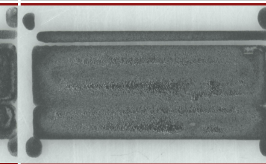
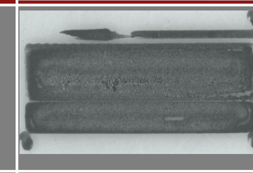
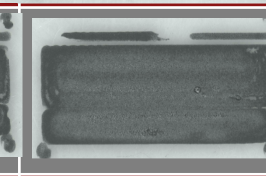
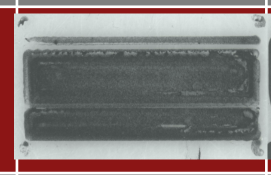
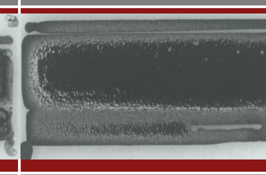
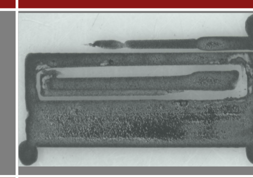
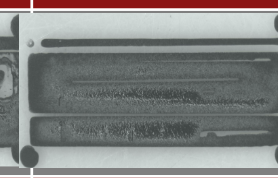
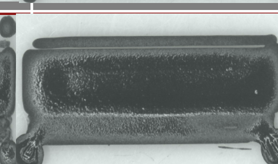
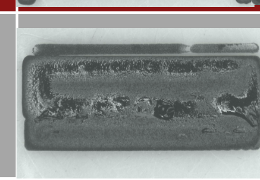
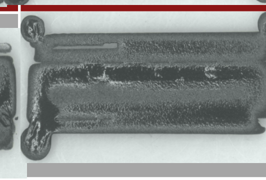


Both inks are shear-thinning

Voltera: Design of experiments





Kick\RS	0.14	0.18
0.05mm		
0.2mm		
0.4mm		
0.6mm		
0.8mm		
1.0mm		

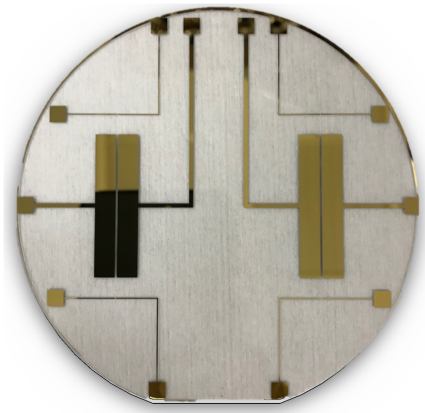
Thicker traces

Homogeneous

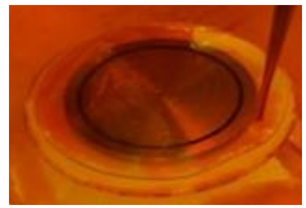
Stanford University

Current Collectors

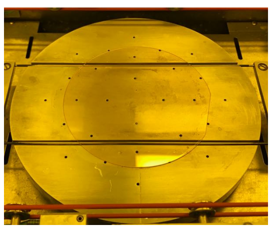
All-Litho	Wbflexcorr	Heidelberg	Svgcoat and dev	Innotec or AJA	Wbflexsolv

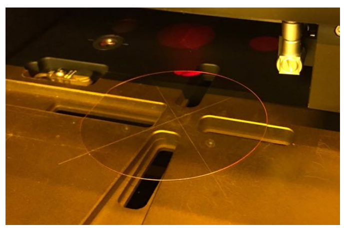


(0) 4” glass wafer (500 um thick). HF 8 seconds → SRD → YES oven.

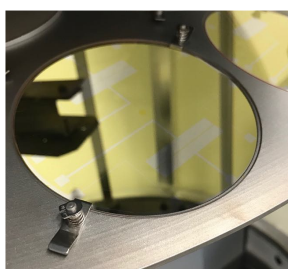


(1) Spincoat LOL-2000 250 nm (***Headway***, 1800 RPM, 1 min) → Hotplate @ 200C, 5 mins → Spincoat Shipley 3612 1 um (***Svgcoat***, recipe # 7).

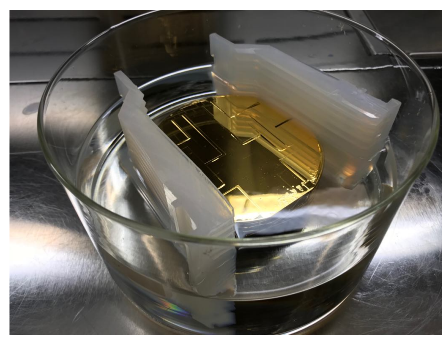




(2) ***Heidelberg*** expose @ 120 mJ/cm^2 dose, -2 defocus → PEB (***Svgdev***: station #1, recipe #9 then station #2, recipe #1) → DEV (***Svgdev***: station #1, recipe #3 then station #2, recipe #1).

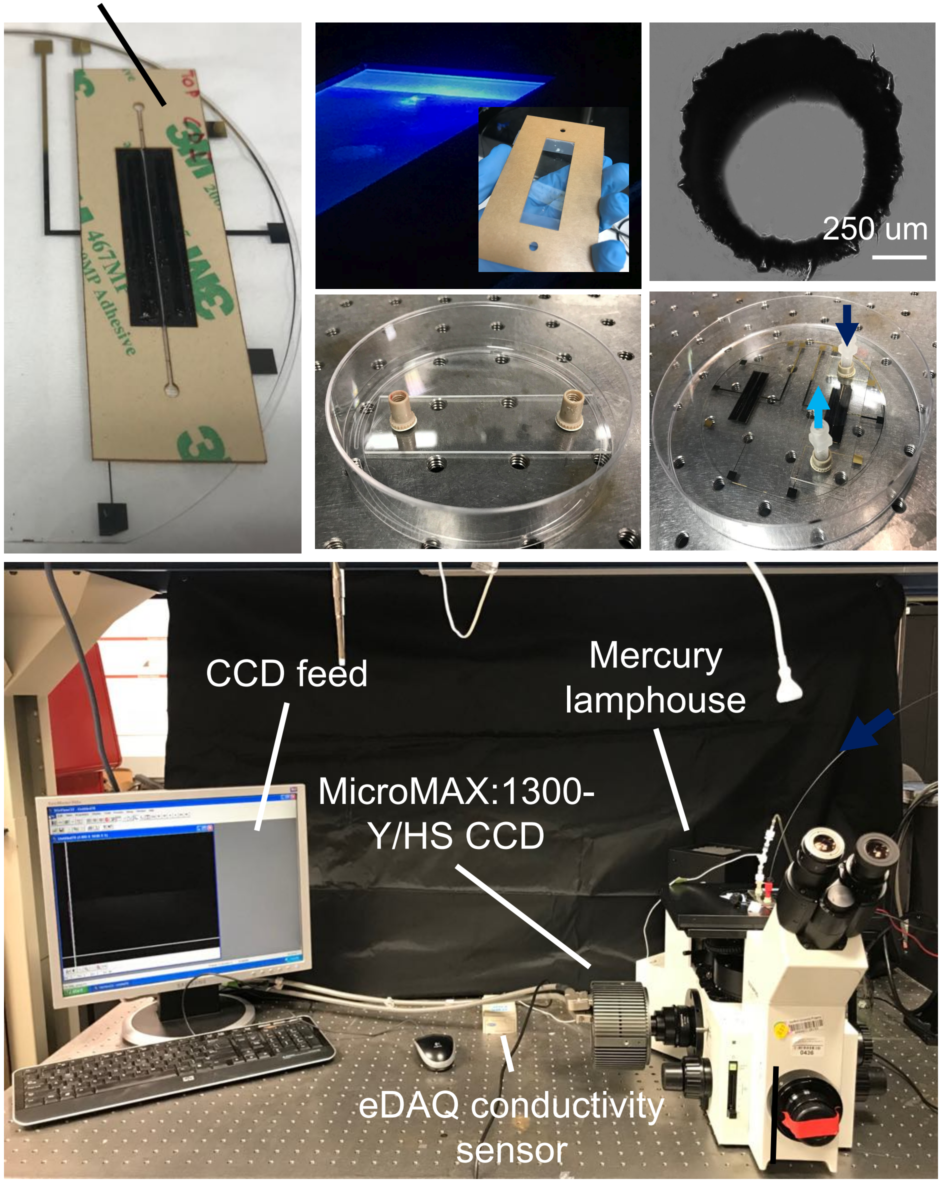


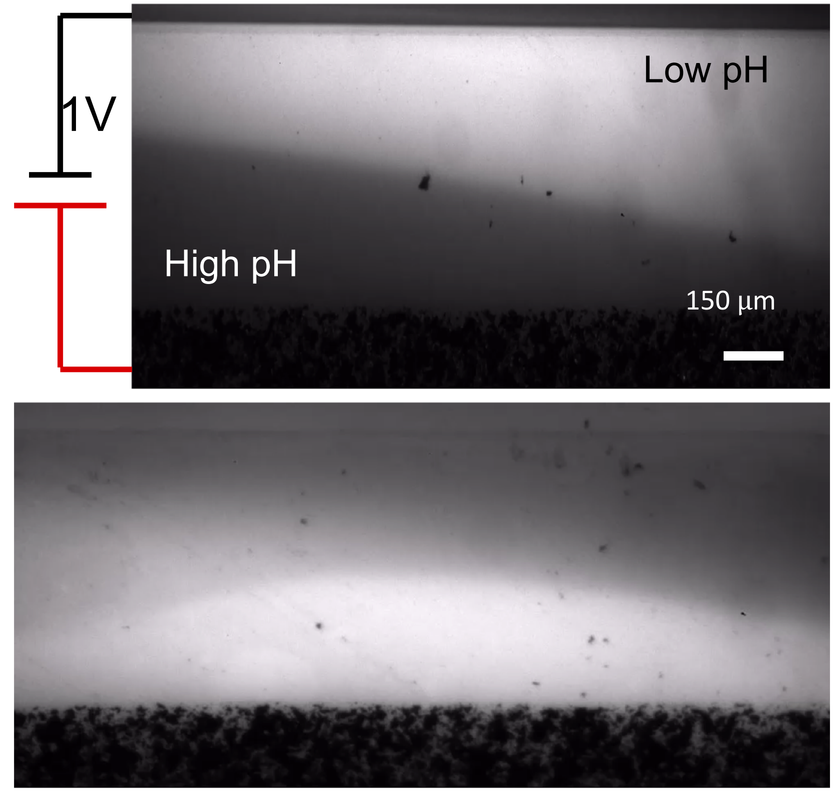
(3) ***Innotec*** E-beam evaporation Cr (2% power, 10 nm) and Au (14% power, 200 nm)



(4) Acetone bath overnight → DEV for LOL removal (***Svgdev***: station #1, recipe #3 then station #2, recipe #1).

Assembly + Flow Test Set-Up+ preliminary visualization





- Laser cut glass, bond IDEX 10-32 ports
- Silicone gasket (+epoxy) → 3M 200MP double-sided adhesive

Takeaway

- In the US, 80% of desalination market is brackish water
- CDI can produce 5x quantity of water for same energy as RO
- Microfabricated CDI to achieve lower contact resistance, tailored geometry, and understanding

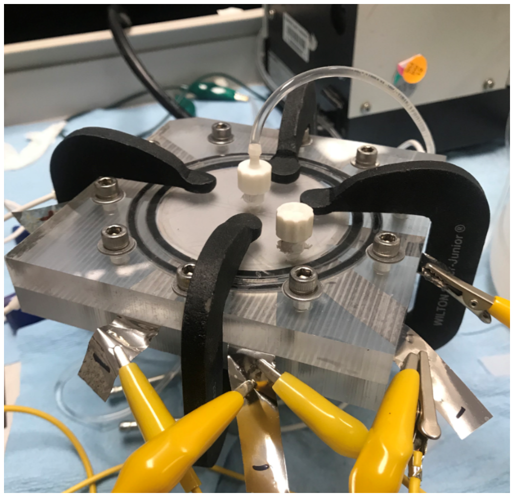
Thank you to all!

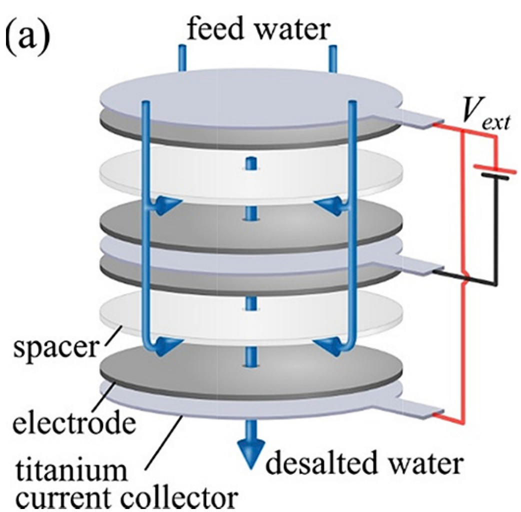
Questions?

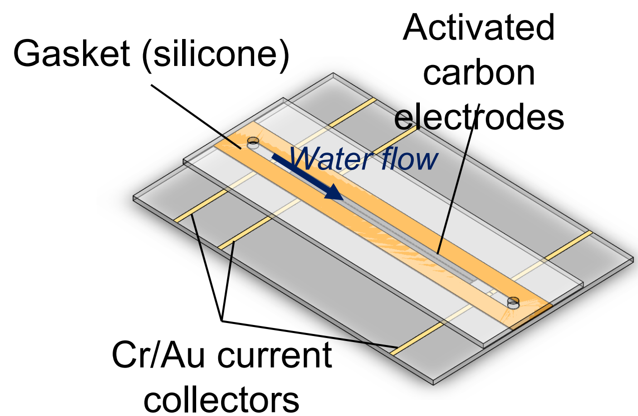
Santiago Group

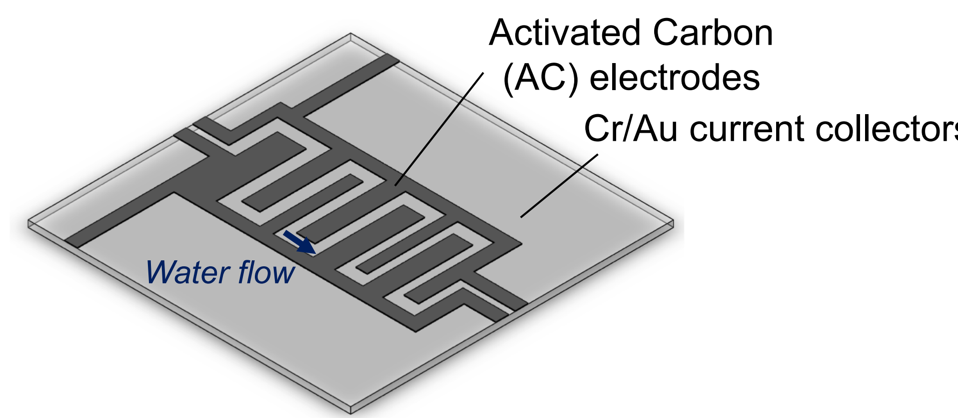
Backup

Current CDI Cell Design and Goal Design







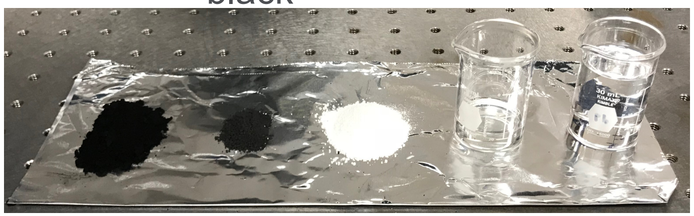


CDI Team – Week 3 Update

(1) Mix slurry components

0.25 g carbon black

6 mL Terpinol

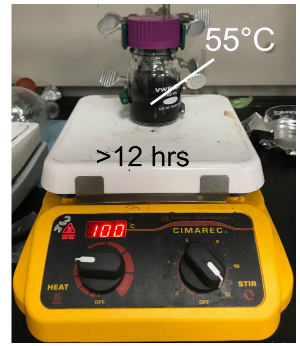


2 g AC

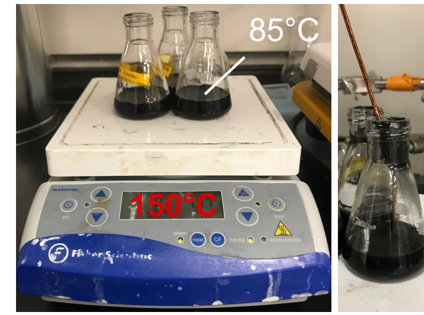
2 g ethyl cellulose

30 mL IPA*

(2) Homogenize solution



(3) Evaporate IPA

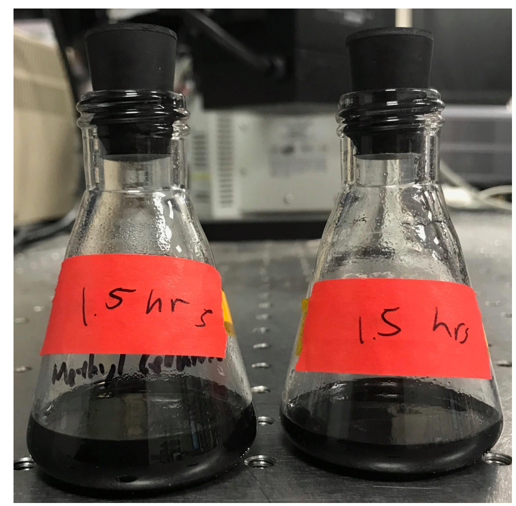


DoE 1.A: Deposition

- Previous: vary (1) IPA content*, (2) *Voltera* kick, and (3) *Voltera* rheological setpoint

- New: fix IPA content, vary (1) evaporation time at 150°C
  - On a hot plate, evaporation rates are inconsistent

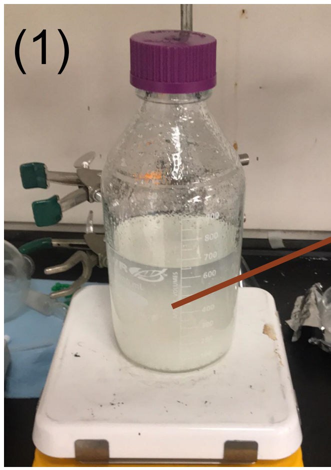
- How to vary viscosity robustly, on-demand?



Difference in evaporation rates for same slurry and evaporation time @ 150°C

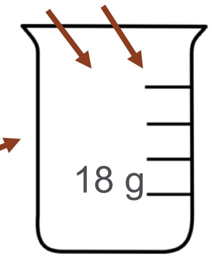
Fast Slurry Prep and IPA % Control

(1)



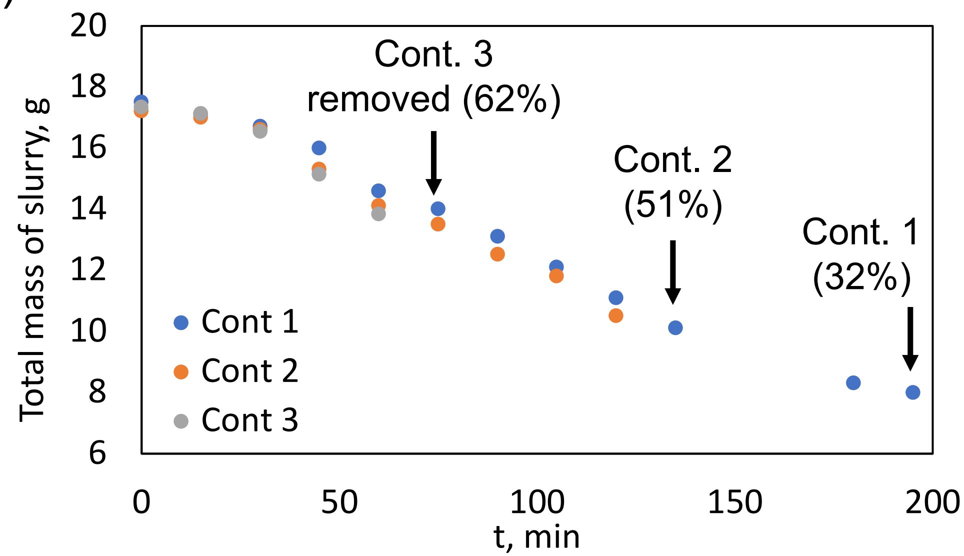
500 mL IPA
100 mL Terpinol
33.3 g Ethyl Cellu.

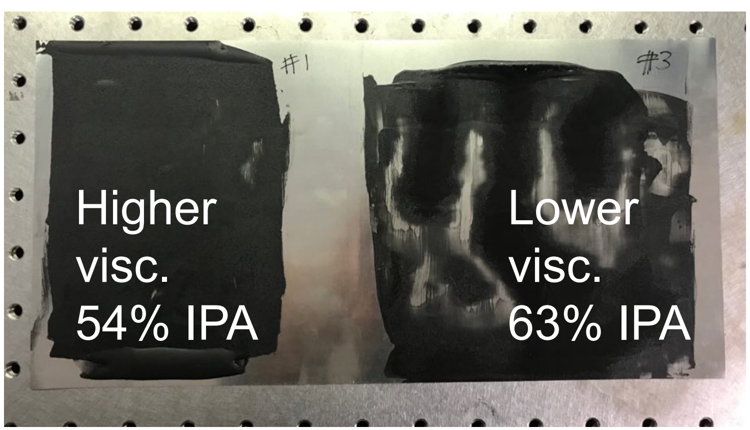
(2) 1 g AC + 0.12 g CB



(3)

(4)

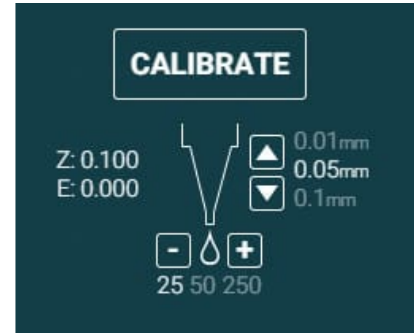


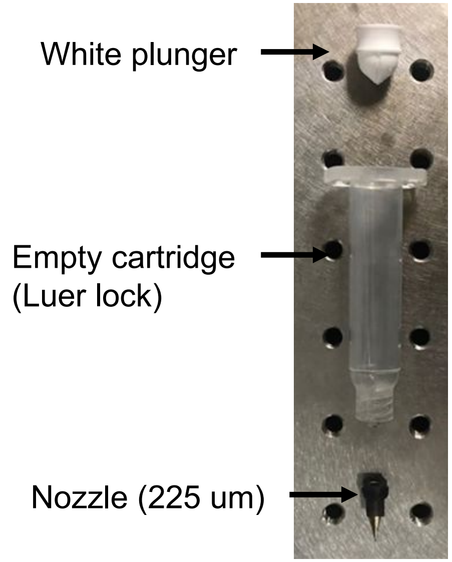


- Control IPA % in AC slurry by heating and measuring mass change
- Viscosity is a function of % of IPA content.
- Ducter blade tests to find range of IPA % (higher viscocity -> better spread)

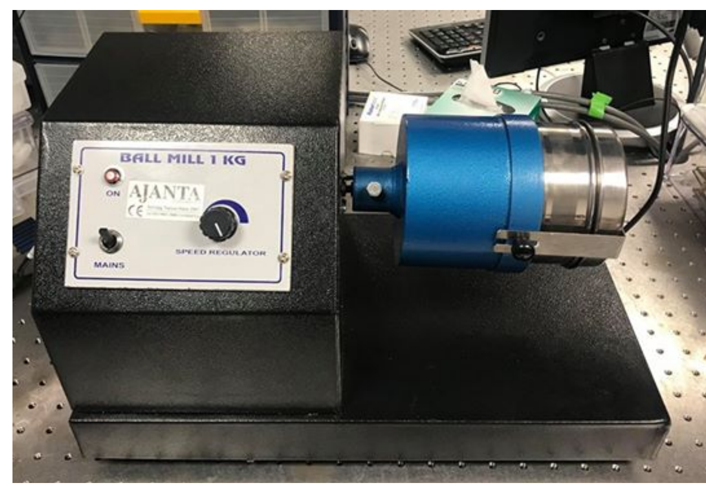
Slurry Loading and Calibration

- Transfer pipette to load custom ink into cartridges
- Nozzle clogged after printing for ~5 mins
  - Size of AC particles
  - IPA evaporation in nozzle
- Real time flow parameter calibration
- Ball mill



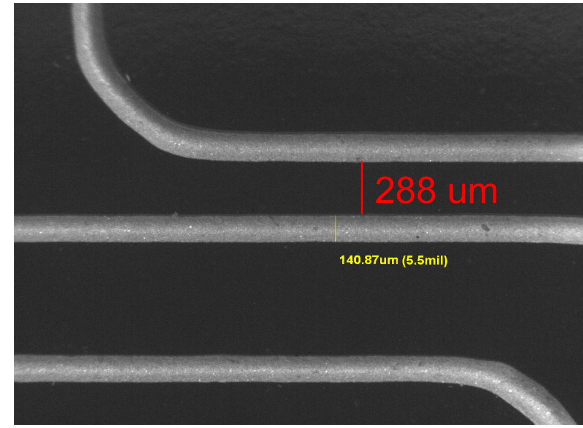


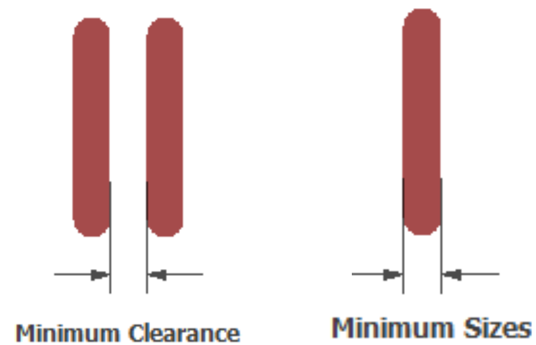
Need to buy ~20x

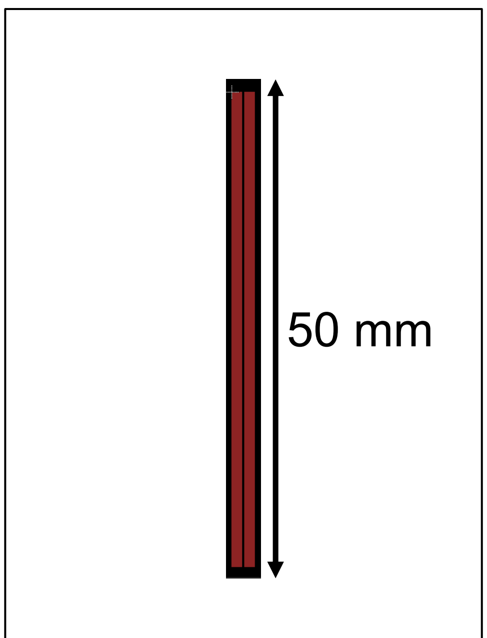


"Circuit" Design – Eagle -> Voltera

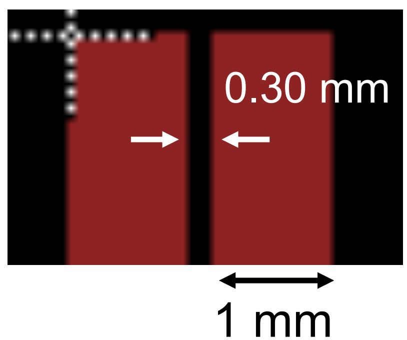
- Voltera CAM Processor
  - Generate gerbers for Voltera software
- Design Rule Check
  - Max board: 100 x 125 mm^2
  - Min trace width/spacing: 0.254 mm

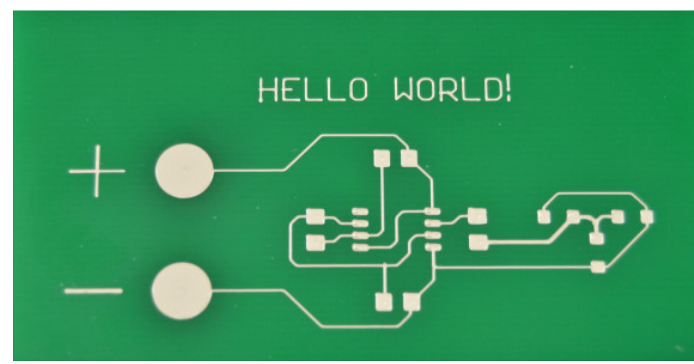






75 x 50 x 1 mm Glass Slide

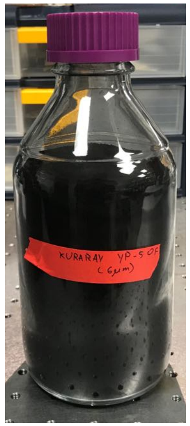




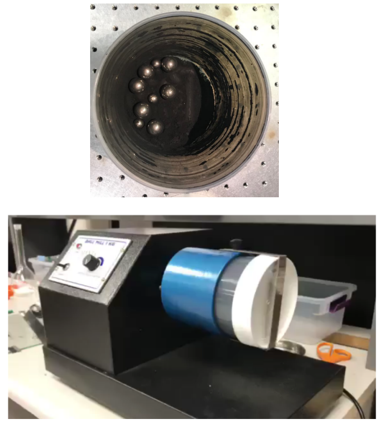
AC Powder – Size Matters (Viscosity Too)

CABOT kuraray

(1)

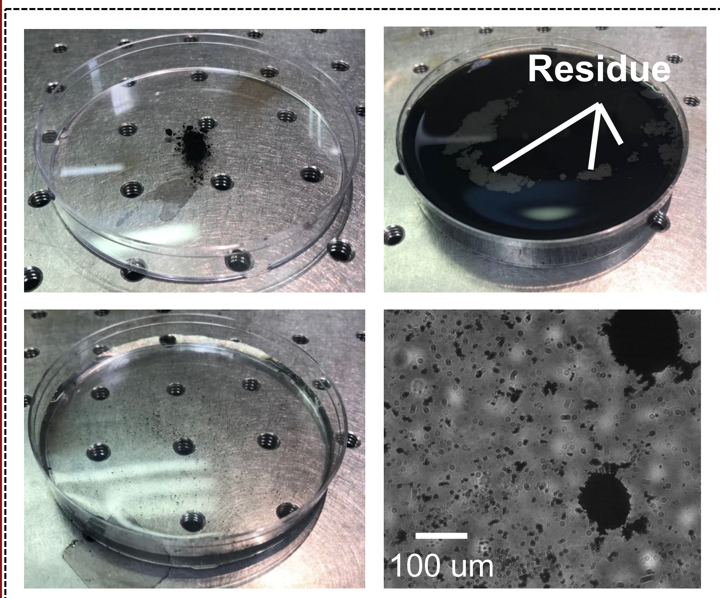


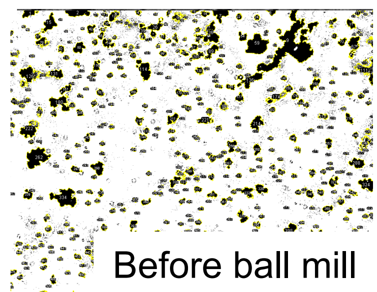
(2)

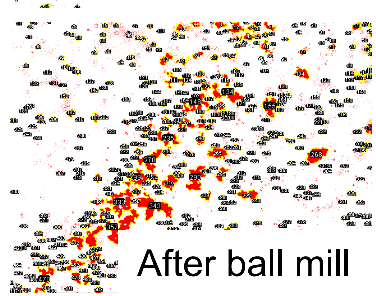


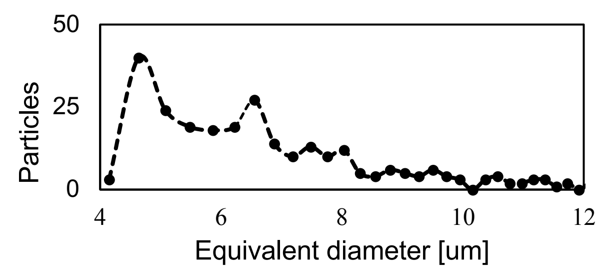
(3)

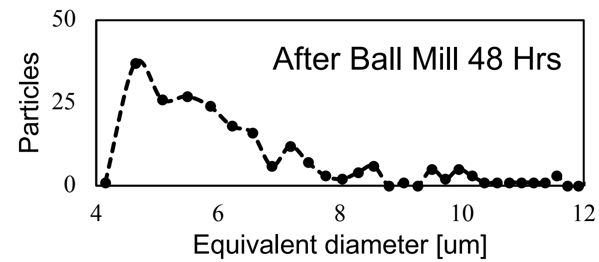
- Activated carbon slurry made from terpinol, ethyl cellulose, IPA, carbon black and **AC powder**
  - 2 AC powder brands -> One held in ball mill 48 hours
- Place AC powder in petri dish, dilute in water, remove *residues*, repeat 3x-5x times











Pass spacing

Controls minimum center-to-center distance between two adjacent lines printed with the V-One.

Dispense height

Distance between the top of the nozzle and the substrate surface during printing. The V-One dynamically corrects the height to maintain this value as constant (+/- about 20um)

Feedrate

XY-axis travel speed during dispensing. Does not affect travel speed when moving between features.

Trim length

Maximum distance the printer will dispense for before it stops and re-pressurizes the cartridge (by applying another 'Kick'). The reason is that pressure within the cartridge decreases slowly while dispensing, so the V-One needs to compensate for the flow rate and re-pressurize between traces.

Trace penetration

To make sure traces and pads connect properly, the V-One plans ahead so that traces will travel a small distance into a pad. This determines how far in the trace goes.

Anti-stringing distance

Depending on the surface energy of a fluid, it might prefer to stick to the nozzle than the board, which produces 'stringing' when the nozzle raises. The V-One adjusts for this by travelling back over the feature to safely break the string, rather than allowing the string to break during raise/travel.

Soft start / stop ratio

Due to compressibility of the fluid and mechanical factors (like speed of the motors) there is a delay between when the Kick is initiated (pressurization starts) and when the flow rate reaches the correct level to start a line or other feature his is similarly true for terminating the flow rate/releasing pressure. These parameters are used to appropriately compensate for this. These don't typically need to be adjusted except for particularly compressible or viscous materials.

Kick

This parameter controls the Stroke Length of the piston within the dispenser, hence how much pressure is applied. This parameter, along with the rheological setpoint, are the most important for printing different fluids. Fluids that are highly compressible and viscous will need a large kick to initiate flow and force the fluid through the nozzle, while lower viscosity fluids need a smaller Kick. Incompressible and low-viscosity fluids will only need a very small Kick to initiate flow.

There is often confusion regarding Kick and the +/- settings - while both control the piston, they do different things. The Kick is the MAXIMUM displacement of the piston, while +/- adjust the relative starting position of the piston.

Rheological setpoint

As mentioned above, this parameter and the Kick are the two most important parameters for printing consistency and quality. While Kick controls the amount of pressure applied to the fluid, Rheological Setpoint controls how the V-One compensates for the flow rate over time. For high-viscosity compressible fluids (like the conductive ink or solder paste), this parameter is increased if the flow rate is decreasing over time.

Atypical use case: Incompressible / low-viscosity fluids:

For incompressible fluids, this parameter is used in a slightly different way - you want to adjust it to account for total fluid volume you want dispensed each stroke. *However*, for reasons to do with the typical use-case and the algorithm, this approach will only work for printing many identical features - meaning if you're printing a circle that is 0.5mm in diameter, vs. one that is 0.3mm in diameter, you'd need to change the rheological setpoint to get the same fluid volume dispensed.

Voltera: Specs

PRINTING	
Minimum Trace Width	0.2mm
Minimum Passive Size	1005
Minimum Pin-To-Pin Pitch	0.65mm